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32692	Patent
Customer Number	Case No.: 58399US002

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	M. Benton Free and Mikhail L. Pekurovsky		
Serial No.:	10/607,698	Examiner:	Frederick John Parker
Filed:	June 27, 2003	Tech Center:	1762
For:	PATTERNED COATING METHOD EMPLOYING POLYMERIC COATINGS (as amended)		

Certificate of Facsimile Transmission	
Pursuant to 37 CFR 1.8, I certify that this correspondence is being sent to the telephone number shown below, addressed to Mail Stop AF, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450, on the below indicated date.	
Facsimile Number 571-273-8300	Signature
Date of Transmission December 16, 2005	Printed Name Lynelle K. Grube

DECLARATION UNDER 37 C.F.R. §131 OF GREGORY F. KING

MS: AF
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

I, Gregory F. King, hereby declare that:

1. I received a B.S. degree in Mechanical Engineering from the University of Minnesota in 1994 and an M.S. degree in Mechanical Engineering from the University of Illinois at Urbana-Champaign in 1997.

2. I have been employed by 3M since 1997. My initial assignment was in the 3M Engineering Systems and Technology Center (now called the 3M Central Research Process Laboratory). I recently transferred from that assignment to the 3M Specialty Film Division Laboratory (now called the 3M Display and Graphics Film Laboratory). Throughout my 3M assignments I have worked on coating technologies. My present title is Coating Technology Specialist.

3. Enclosed with this Declaration is a copy of 3M Record of Invention ("ROI") No. N005214 entitled "A method of patterning in coating". The signature dates and times at the bottom of page 1 have been covered on the enclosed ROI. The original ROI is in color.

In all other respects the enclosed ROI is a true and accurate black and white copy of the original ROI.

4. The ROI was written by my 3M colleague Mikhail L. Pekurovsky. I worked near Mr. Pekurovsky when the work he described in the ROI was performed, and I had first-hand familiarity with that work through personal observation.

5. Prior to April 24, 2002, I read and understood and signed the ROI as a witness. I did so by reviewing the ROI using a 3M-developed electronic ROI creation and recordkeeping system that runs within Lotus Notes. I obtained access to the system by logging in using my 3M-assigned individual Lotus Notes user id and password. I read the ROI onscreen and signed it electronically by clicking an onscreen button confirming that I had read and understood the ROI. The date and time of my signature was recorded electronically and affixed to the original ROI at the bottom of page 1 under my printed name.

6. The ROI describes a method for preparing a patterned article, including steps of applying a release polymer (a fluoropolymer) to a portion of a glass substrate in a desired pattern (a slightly-curved stripe), applying a continuous layer of a substrate-adherent polymer (a polyimide) over the pattern and over at least a portion of the substrate, applying an adhesive tape to the polyimide, and removing the adhesive tape and polyimide adhered to the tape atop the pattern while leaving a portion of the polyimide adhered to the substrate in a negative of the pattern. Figure 2 on page 5, Figure 3 on page 6, Figure 4 on page 7 and Figure 5 on page 8 confirm these steps.

7. I know from my familiarity with Mr. Pekurovsky's work relating to the ROI and from personal experience with the materials he employed in that work that the fluoropolymer Mr. Pekurovsky used as a release polymer has a surface energy less than that of the polyimide Mr. Pekurovsky used as a substrate-adherent polymer.

8. The ROI also describes a method in which the continuous layer of polyimide substrate-adherent polymer has a substantially constant height with respect to the glass substrate. Figure 2 on page 5 illustrates this feature. Figure 5 on page 8 (which shows a height of about 0.23-0.29 μm for the polyimide layer) and Figure 6 on page 9 (which shows a height of about 0.28-0.29 μm for the polyimide layer) confirm it.

9. The ROI also describes a method in which the substantially constant height with respect to the glass substrate is less than about 15 μm. Paragraph 4 on page 3 (which says that "Polyimide coating was spin coated at 6000 rpm and was around 0.3 to 0.4 micrometers thick"), Figure 5 on page 8 and Figure 6 on page 9 confirm this step.

10. The ROI also describes a method in which the substantially constant height with respect to the glass substrate is less than about 3 μm. Figure 5 on page 8 and Figure 6 on page 9 confirm this step.

11. The ROI also describes a method in which the substantially constant height is about 2 to about 10 times the release polymer pattern thickness. Paragraph 4 on page 3 (which says that "Thickness of the fluoroplymer layer was measured by Tenkor profilometer and appears to be around 0.5 micrometers when deposited at a low speed and around 0.1 micrometers when deposited at a higher speed. Polyimide coating was spin coated at 6000 rpm and was around 0.3 to 0.4 micrometers thick.") and Figure 5 on page 8 (which shows a height of about 0.23-0.29 μm for the polyimide layer and a height of about 0.09 μm for the fluoropolymer layer) confirm this step.

12. The ROI also describes a method in which after removing the tape the pattern has at least one sidewall the major exposed portion of which is substantially perpendicular to the substrate. Figure 5 on page 8 (which shows that the exposed polyimide sidewalls are perpendicular to the glass substrate) confirms this step. Figure 6 on page 9 (which shows the exposed polyimide sidewalls after the fluoropolymer layer has been removed using hydrofluoroether solvent) provides further confirmation.

13. The ROI also describes a method in which the patterned article is a printed circuit board, electrical connector, information display or electronic component. Paragraph 3 on page 2 (which says that "In many applications, especially that involve electronics, it is necessary to have certain parts of substrate to be without coating, or, in other words, to have coating layer patterned. An obvious example is an electric contact that needs to be clear of coating for later assembly.") confirms this step.

14. All statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true; and further that these statements were

made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the Application or any patent issuing thereon.

Further Declarant saith not.

12/16/05
Date

Gregory F. King

enc. 3M Record of Invention No. N005214 (with signature dates and times covered)

3M Confidential

Engineering Manufacturing and Logistics
Record of Invention

Attorney/Client Privileged Information

ROI Number Assigned by Engineering Manufacturing and Logistics :	**N005214** **EML-02191**

Title:	A method of patterning in coating

3M Investigator(s) Information:

☐ None Existing

Name (First, Middle Initial, Last)	3M Employee No.	3M Dept. Name	3M Dept. No.	Signature
MIKHAIL L PEKUROVSKY	01336343	ESTC - PP&ML	600463	
MICHAEL B FREE	01342955	ESTC - APS	600461	

Non-3M Investigator(s) Information:

[X] None Existing

Organization Name	Investigator Name (First, Middle Initial, Last)

Signature Information:

Described by:		This document has been read and understood by me.	
Signature:	Electronically signed	Witness Signature:	Electronically signed
Print Name:	MIKHAIL L PEKUROVSKY	Print Name:	GREGORY F KING
Date:	Date and Time Redacted	Date:	Date and Time Redacted

A method of patterning in coating

Introduction

In spin coating a liquid layer on the top of a substrate is thinned by centrifugal forces when that substrate is rotated at a couple of thousands of revolutions per minute. On uniform substrates spin coating produces coating layer of uniform thickness when liquid rheology, air flow, and substrate topology, among other parameters, cooperate.

There are other methods available to deposit liquid layers onto solid substrates. They include dip coating, when substrate is withdrawn from a pool of liquid entraining thin liquid layer with it; die coating, when liquid is extruded from a slot onto the substrate; roll coating, when liquid is deposited onto the substrate in a small gap between two rotating rollers; spray coating, when liquid is atomized into small droplets that attach to the substrate; and many others.

In many applications, especially that involve electronics, it is necessary to have certain parts of substrate to be without coating, or, in other words, to have coating layer patterned. An obvious example is an electric contact that needs to be clear of coating for later assembly.

In spin coating, available methods to make patterns include masking with tape and photoimaging. In masking, a tape is first applied over parts of the substrate where no coating is desired. After spin coating and drying the tape is removed to expose areas of the substrate that it covered. Downsides of using tape for masking are coating build-up near tape edges and complication involved in applying and removing the tape. In photoimaging the liquid is chosen to be curable only during exposure to radiation, usually in the ultra-violet spectrum. After spin coating deposits a layer of liquid on the substrate a mask is positioned over the surface of the coating and coating is exposed to radiation only where the mask has openings. After curing under exposure, the substrate is put in a solvent bath that removes the resin that remains uncured, patterning the coating. Downsides of photoimaging are limited choices of resins and need to use strong solvents for removing uncured resin.

Concept of the Invention

In described a thin layer of a polymer that is not wet by a coating liquid or does not adhere to cured coating is first deposited on the areas that are intended to stay free of coating. Fluorinated polymers can be successfully used for that purpose. In most of the cases polymer layers should be thinner than final coating thickness, but in certain applications, thicker than coating layers can be beneficial.

Arguably, when polymer layers are thicker than intended coating thickness the mechanism by which the present invention works is related to instability of thin liquid films. Stability of thin liquid layers is to some extent related to wetting properties between liquid and substrate. When liquid wets the substrate, thin liquid layers are stable. Contrarily, when liquid does not wet the substrate, thin liquid layers are unstable and liquid dewets from the substrate. During spinning or other coating procedure, liquid layer thins and eventually becomes unstable over the areas covered with, for example, a fluoroplymer. Under the action of dewetting forces (as well as centrifugal forces in spin coating or gravity forces in dip coating) liquid is completely removed from areas covered with fluoropolymer. After coated layer dries and cures the layer of non-wetted polymer is removed with appropriate solvent.

In spin coating, because polymer thickness is larger than the coating thickness, the polymer layer can significantly disturb air flowing outward from that layer and, as a result, lead to coating non-uniformity. Small liquid drops emanating from the top of the polymer ridge also can cause coating non-uniformity. When areas outward of polymer covered areas are of no importance, coating distortions there can be tolerable.

When polymer layer thickness is lower than intended coating thickness, coating liquid covers the top of the polymer layer during spin coating. Thin layer of coating liquid can dry on the top of the polymer layer without dewetting. After coated layer is cured, part of it can be removed or delaminated from areas covered with polymer by mechanical means, for example, with a strong adhesive tape. The polymer layer can be later removed with an appropriate solvent.

A Realization of the Invention/Supporting Experiments

Spin coating

Experiments were performed with polyimide coating and fluoropolymer as a polymer that was deposited on the areas that were intended to stay free of polyimide. Glass slides, two by three inches in size, were used for the experiment. Fluoropolymer was deposited with a cotton swab moved by hand at a low and at a high speed. Thickness of the fluoroplymer layer was measured by Tenkor profilometer and appears to be around 0.5 micrometers when deposited at a low speed and around 0.1 micrometers when deposited at a higher speed. Polyimide coating was spin coated at 6000 rpm and was around 0.3 to 0.4 micrometers thick.

I. Thick polymer layer

Figure 1 shows schematic of using thick fluoropolymer layers to mask areas near edges. During spin coating polyimide solution dewetts from areas covered with fluoropolymer. After coating the fluoropolymers is removed with solvent.

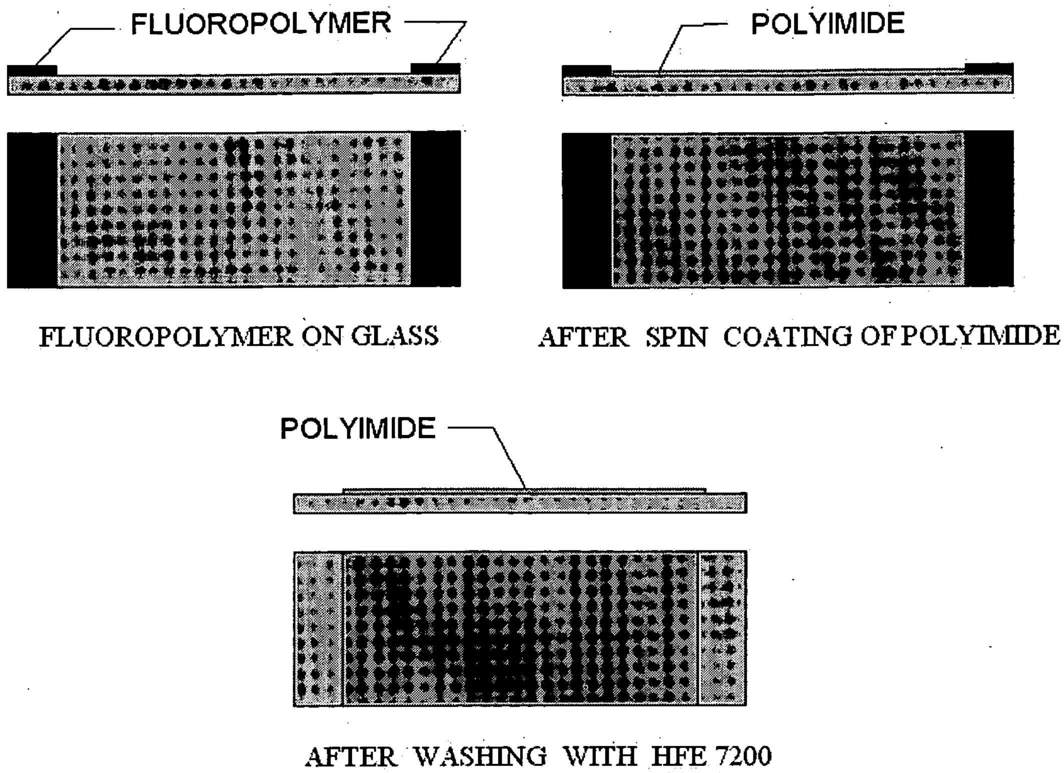

Figure 1 Masking with thick layer of fluoropolymer in spin coating.

II. Thin polymer layer

Figure 2 presents a glass slide with deposited fluoropolymer lines and coated with polyimide. In certain areas polyimide accidentally delaminated during handling. Figure 3 shows a profile of fluoropolymer layer before polyimide layer was deposited.

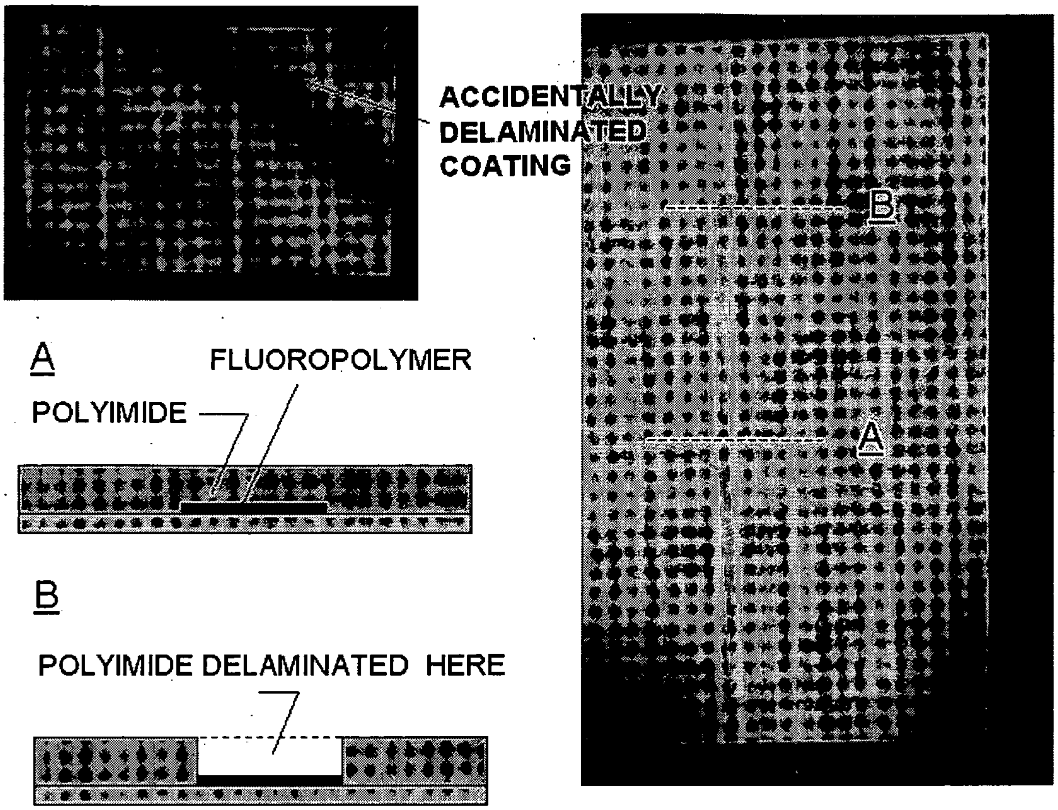

Figure 2 Glass slide with deposited fluoroplymer lines and spin coated with polyimide. Top left image shows the whole slide which is two by three inches in size. Sections of the polyimide coating accidentally delaminated during handling.

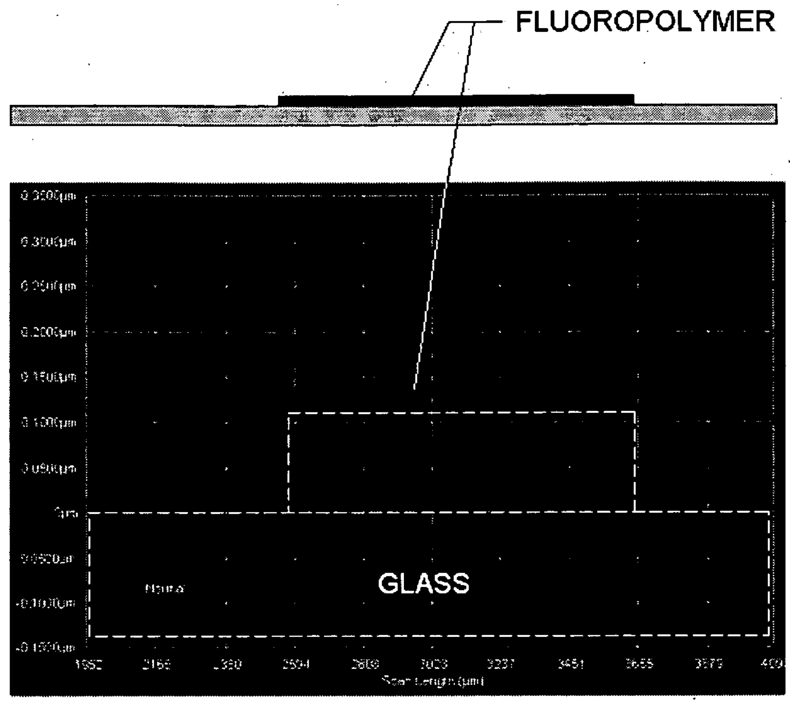

Figure 3 Profile of a fluoropolymer layer. The fluoroplymer layers were deposited with a fastly moving cotton swab and are about 0.1 micrometers thick.

After polyimide layer was deposited and dried, an adhesive tape was applied on the top of the polyimide layer. During removal of the tape polyimide layer delaminated from the areas covered with fluoropolymer, Figure 4 and Figure 5. Fluoropolymer was later washed from the glass surface by solvent HEF 7200, Figure 6.

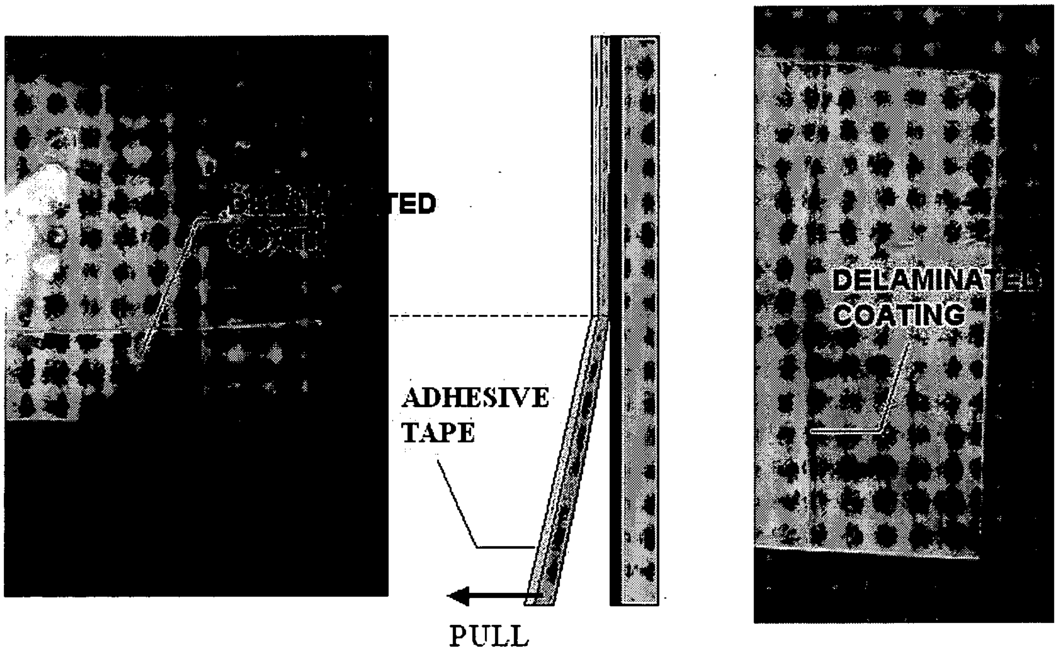

Figure 4 Process of delamination of polyimide coating with adhesive tape. Polyimide layer delaminates only from areas covered with fluoropolymer.

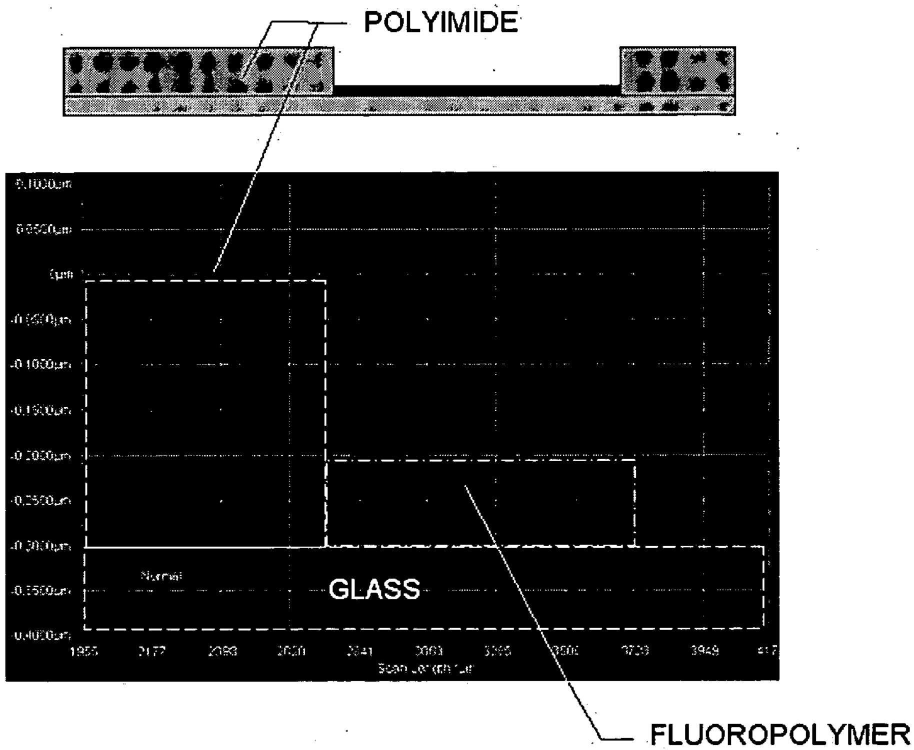

Figure 5 Profile of the area covered with fluoropolymer after delamination of the polyimide from the top of the fluoroplymer line.

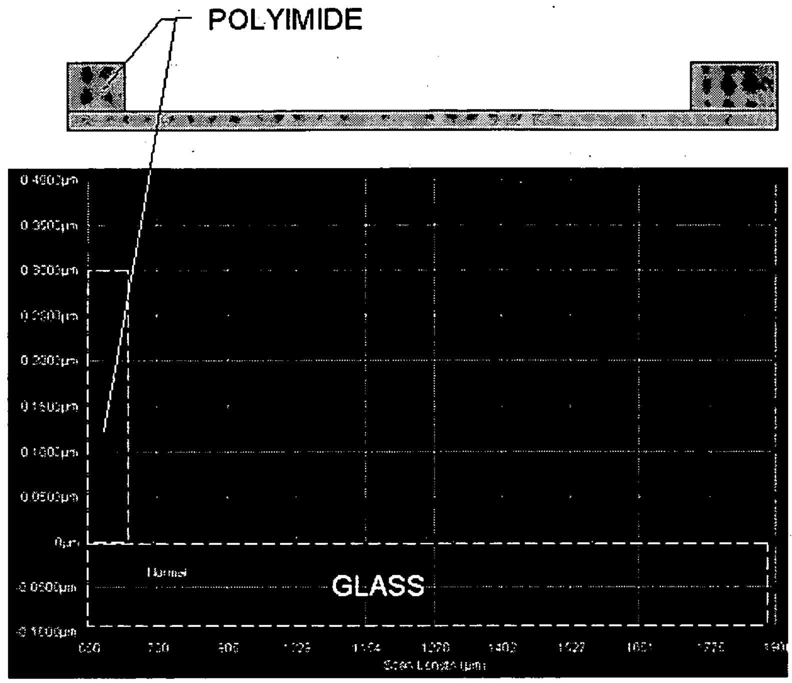

Figure 6 Profile of the area with delaminateded polyimide and fluoropolymer washed out by HFE 7200

Dip coating

Similar technique was used in dip coating, Figure 7. A spot of fluoropolymer was deposited on a glass slide, glass slide was submerged in a solvent-based ink and withdrawn at a low speed. Withdrawal of the glass slide left a thin layer of ink on its surface. After drying of the ink layer, ink atop of the fluoropolymer spot was delaminated with an adhesive tape. Figure 8 shows profile of the spot area after delamination and after washing the area with the appropriate solvent, HFE 7200. Profiles show clean delaminated boundary and no distortion of coating thickness near the fluoropolymer spot.

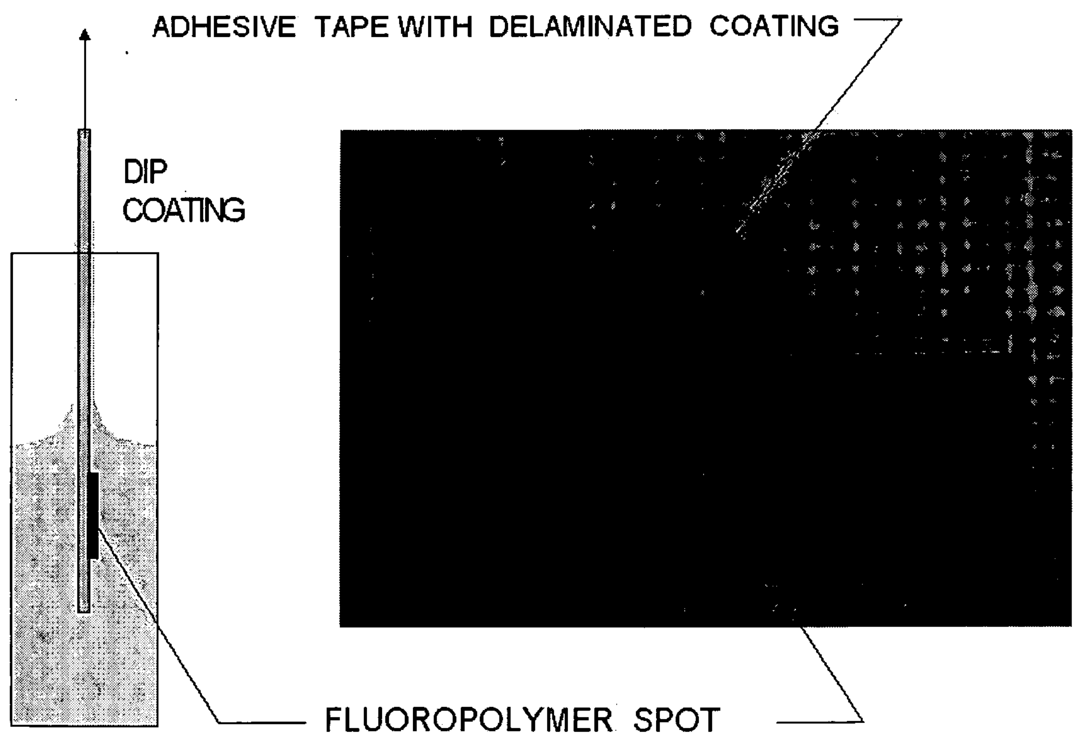

Figure 7 Patterning in dip coating. Blue ink delaminated from fluoropolymer coated spot when an adhesive tape was applied and then removed from the surface.

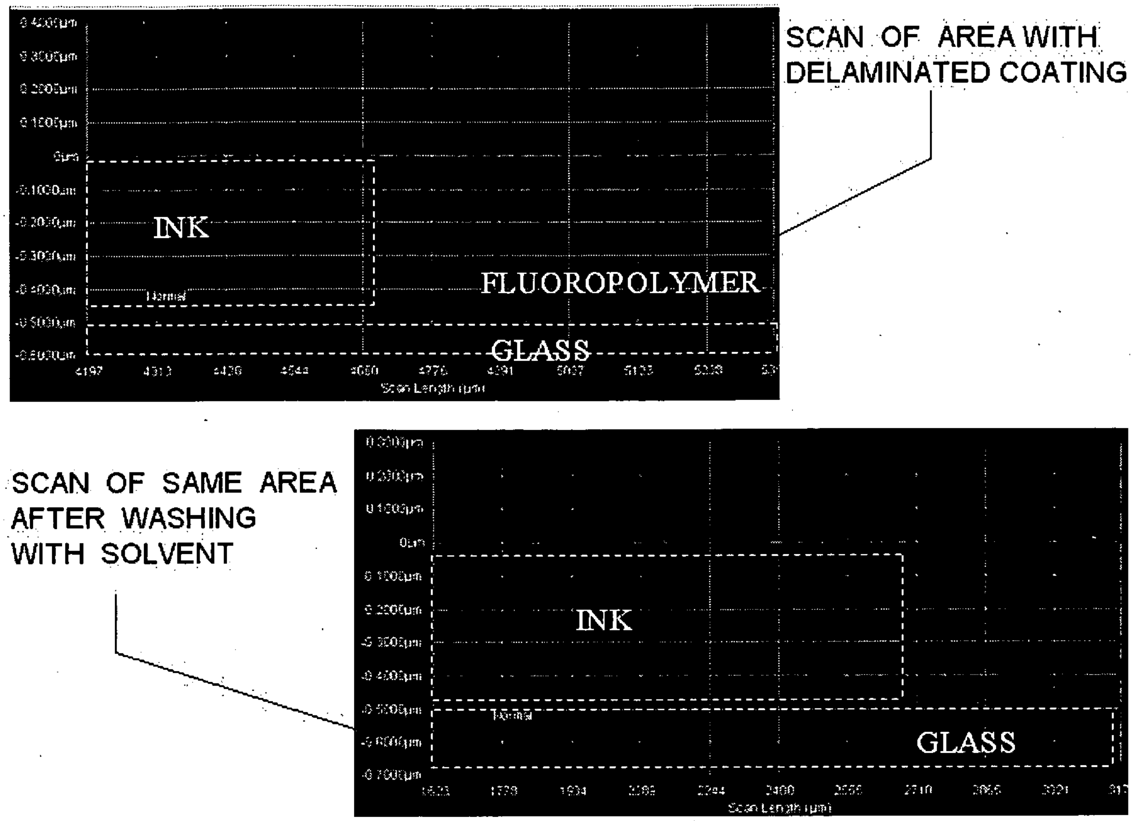

Figure 8 Profilometer scans of the neighborhood of the area covered with fluoropolymer, dip coated with ink right after the ink layer was delaminated from the top of the fluoropolymer with help of an adhesive tape. Bottom image shows scan of the same area after washing it with HFE 7200 solvent.

Significant Features or Advances

1. A method of patterning in coating which involves depositing layer of polymer that coating liquid does not wet on the areas that are intended to stay free of coating.
2. Item 1 applied specifically to spin coating
3. Item 1 applied specifically to dip coating
4. Item 1 with non-wetting layer thinner than intended coating
5. Item 3 with polyimide layer removed from atop of non-wetting areas by adhesive tape
6. Item 3 with polyimide layer removed by other means
7. Item1 with non-wetting layer thicker than intended coating.
8. Items 5,6, and 7 with non-wetting layer removed by an appropriate solvent after coating layer is delaminated